Report No. IITRI-C6030-6
(Progress Report)

BACKSTREAMING FROM OIL DIFFUSION PUMPS

National Aeronautics
and Space Administration

IIT RESEARCH INSTITUTE

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I. INTRODUCTION

The objective of this program is to evaluate the factors contributing to backstreaming contamination in modern, well baffled, oil diffusion pump systems. The combinations being studied consist of fractionating and non-fractionating pumps, one bounce, right angle elbow and two bounce chevron baffles, and the pump oils DC 705, Convalex 10, and OS 124.

During this report period we have succeeded in reducing backstreaming contamination to amounts much less than previously and have found that, under the optimum conditions, there is very little difference between the various pumps, baffles, and oils. Two baffles, the NRC Cryo baffle and the Granville-Phillips have not performed as well, however, further conditioning may reduce the backstreaming.

A second objective of the program is to measure contamination from turbo-molecular pumps. These pumps have been received and are being setup.

II. EXPERIMENTAL WORK - OIL DIFFUSION PUMPS

During recent work very low backstreaming values have been consistently obtained for three out of five test stations. This is a direct result of experience with the system and conditioning of the oil. These low results have required a re-examination of our collection and analysis techniques. Consequently a standard procedure as listed below for conducting the runs has been established in an attempt to minimize the variation in data when working with such small quantities of oil.

1. Start-up diffusion pump with 50% of rated heat input for 1-1/2 hours.
2. Cooling the outside of chevron and HN-6 baffles.
3. Bakeout of collection plate (with baffle cold) for 18 hours.
4. Run time should be at least 70 hours.
5. System should be kept dry during removal of sample.
6. System pressure should be 2×10^{-8} torr or less (LN_2 trapped gage).

A. Rate of Heating on Startup

Previous data (Report C6030-5, Table 3, runs 93, 95, 96) on Station 5 using Convalex 10 and without bakeout of the collection plate at the start of a run indicated a minimum startup deposit when 50% of rated heat input was used in starting up the diffusion pump. An adequate bakeout procedure should eliminate any differences from startup effects, and this is generally true of recent data using steam heating of the collection plate at the start of the run. However, even after such bakeouts (see C below) measurable deposits are always present. Consequently, 50% of rated heat input on startup was adopted as a standard procedure. This involves very little extra effort and the pump performance, as indicated by the system pressure, is not noticeably affected.

B. Cooling External Walls of Baffles

Previous results (Report C6030-5, Table 4, runs 98, 101, 108) with the BC-61 chevron baffle with DC 705 has shown that cooling the outer walls to 14°F gives a marked decrease in backstreaming values. Table 1, retabulating the results to include total deposit weight, shows that there is a similar effect for Convalex 10. The deposit weight is a more valid criterion since the present low values are relatively independent of run time (see section C). Presumably, the higher values with uncooled walls are due to oil migration along these warm surfaces. Consequently in our standard runs the outer walls of the traps, BC-61 and HN-6, are cooled to 14°F, the maximum available effect with -75° refrigeration and good insulation. Both the right angle baffle and the Granville-Phillips traps as normally used do not have any warm surfaces which could lead to oil migration.

C. Bakeout of the Collection Plate and Run Time

In order to further confine the measurement of contamination to the steady state run period and eliminate startup effects,

Table 1

EFFECT OF COOLING OUTSIDE WALLS OF CHEVRON BAFFLES

Run	Station	Oil	Run Hours	Temp. of Baffle Walls, °F	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2	Backstreaming Rate, $mg/(cm^2)(min)$
109	1	Convalex 10	169	80	1×10^{-7}	7×10^{-8}	1.4×10^{-4}	$.015 \times 10^{-6}$
117			74	14	7×10^{-8}	6×10^{-9}	$.8 \times 10^{-4}$	$.024 \times 10^{-6}$
121			74	14	8×10^{-8}	7×10^{-9}	$.8 \times 10^{-4}$	$.025 \times 10^{-6}$
98	2	DC 705	74	88	5×10^{-8}	3×10^{-8}	$19. \times 10^{-4}$	$.43 \times 10^{-6}$
101			71	80	5×10^{-8}	3×10^{-8}	$18. \times 10^{-4}$	$.43 \times 10^{-6}$
108			264	14	6×10^{-9}	2×10^{-9}	6.5×10^{-4}	$.04 \times 10^{-6}$

bakeout of the collection plate with the trap kept at -75°F was employed. The results are given in Table 2. It is seen that using steam or hot air at 470°F has the same effect and reduces the deposit weight appreciably and, under these optimum conditions, the weight is essentially independent with run times varying from zero to 293 hours. The values with DC 705 are much larger than those with Convalex 10 or OS 124. However, if the analytical blank (see D below) is considered, the net deposit weights decrease to 1×10^{-4} mg/cm^2 $\pm$ 100%. Run 151 which has employed a 470°F bake gave the lowest value and, considering the analytical blank, is essentially zero. The value of 1×10^{-4} mg/cm^2 for the total deposit corresponds very closely to a film thickness of 10^{-7} cm. This latter value is extremely close to the values of $.99 \times 10^{-7}$ and 1.09×10^{-7} cm which are the molecular diameters of Convalex 10 and DC 705, respectively, calculated from

$$\Delta = 1.329 \times 10^{-8} \, (M/\rho)^{1/3} \text{ cm} \qquad \text{(Ref. 1)}$$

Thus it appears that an oil film, over and above any residual material left by the solvent methanol rinse, in an amount equivalent to a monomolecular film is deposited in the early stages of the run. This deposit does not appear to be removed by heating the plate to 470°F in the case of DC 705. In the case of Convalex 10 this deposit was not removed by steam and was removed at 470°F.

(Ref. 1) Dushman, S. and Lafferty, J. M., "Scientific Foundations of Vacuum Technology," 2nd ed., John Wiley and Sons, Inc., 1962.

Table 2

THE EFFECT OF BAKEOUT OF COLLECTION PLATE AND RUN TIME

Run	Station	Oil	Bakeout Temp., °F	Run Hours	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2: Total	Deposit Weight, mg/cm^2: Anal. Blank	Backstreaming Rate, $mg/(cm^2)(min)$: Total	Backstreaming Rate, $mg/(cm^2)(min)$: Less Anal. Blank
139	1	Convalex 10	212	0	-	-	$.7 \times 10^{-4}$	-	-	-
151	1	Convalex 10	470	0	-	-	$.4 \times 10^{-4}$	$.3 \times 10^{-4}$	-	-
114	1	Convalex 10	None	73	6×10^{-8}	9×10^{-9}	1.6×10^{-4}	-	$.052 \times 10^{-6}$	-
152	1	Convalex 10	None	72	3×10^{-7}	6×10^{-9}	1.4×10^{-4}	$.05 \times 10^{-4}$	$.033 \times 10^{-6}$	$.032 \times 10^{-6}$
117	1	Convalex 10	212	74	8×10^{-8}	6×10^{-9}	$.8 \times 10^{-4}$	-	$.024 \times 10^{-6}$	-
137	2	DC 705	212	0	-	-	2.0×10^{-4}	-	-	-
142	2	DC 705	470	0	-	-	2.6×10^{-4}	-	-	-
143	2	DC 705	470	0	-	-	1.9×10^{-4}	-	-	-
104	2	DC 705	None	70	3×10^{-8}	2×10^{-8}	5.0×10^{-4}	-	$.15 \times 10^{-6}$	-
118	2	DC 705	212	74	6×10^{-9}	1×10^{-9}	1.9×10^{-4}	-	$.043 \times 10^{-6}$	-
161	2	DC 705	212	74	9×10^{-9}	1×10^{-9}	2.5×10^{-4}	$.8 \times 10^{-4}$	$.057 \times 10^{-6}$	$.040 \times 10^{-6}$
107	2	DC 705	None	151	2×10^{-8}	2×10^{-8}	5.0×10^{-4}	-	$.056 \times 10^{-6}$	-
153	2	DC 705	212	146	4×10^{-9}	7×10^{-10}	3.2×10^{-4}	$.5 \times 10^{-4}$	$.036 \times 10^{-6}$	$.031 \times 10^{-6}$
149	2	DC 705	212	145	4×10^{-9}	9×10^{-10}	3.2×10^{-4}	2.2×10^{-4}	$.036 \times 10^{-6}$	$.011 \times 10^{-6}$
108	2	DC 705	None	264	6×10^{-9}	2×10^{-9}	6.4×10^{-4}	-	$.045 \times 10^{-6}$	-
157	2	DC 705	212	293	5×10^{-9}	5×10^{-10}	1.9×10^{-4}	$.8 \times 10^{-4}$	$.011 \times 10^{-6}$	$.007 \times 10^{-6}$
172	2	DC 705	212	292	5×10^{-9}	8×10^{-10}	3.2×10^{-4}	$.6 \times 10^{-4}$	$.018 \times 10^{-6}$	$.009 \times 10^{-6}$
158	5	OS 124	None	73	9×10^{-9}	3×10^{-9}	1.3×10^{-4}	$.4 \times 10^{-4}$	$.033 \times 10^{-6}$	$.024 \times 10^{-6}$
165	5	OS 124	212	75	1×10^{-8}	4×10^{-9}	$.7 \times 10^{-4}$	$.3 \times 10^{-4}$	$.016 \times 10^{-6}$	$.010 \times 10^{-6}$
141	5	OS 125	212	75	1×10^{-8}	5×10^{-9}	$.5 \times 10^{-4}$	-	$.015 \times 10^{-6}$	-

D. Analytical Treatment of Samples

In previous runs with greater backstreaming rates it was found that a single rinse with solvent removed 96% of the oil. With the low backstreaming rates presently obtained under optimum conditions the amount of oil was only 20% or less of the previous values. In addition the analytical technique has definite limitations although it has been extended an order-of-magnitude during this program. We have evaluated our sampling procedure for these smaller amounts of oil. Typical data are given in Table 3. These data indicate that after the original sample is removed, a residual analytical blank remains which is relatively constant for any number of successive multiple rinses. This blank varies from .001 to .018 mg for polyphenyl ethers and .020 to .039 for DC 705. Present sampling procedures now call for multiple solvent rinses to follow every backstreaming sample so as to minimize residual oil. Additionally a final sample is taken which can be used as an analytical blank and indicates the over-all accuracy of the analysis. The present values under optimum conditions appear to be approaching the limit of the analytical method. There are, however, still definitive differences between the measured amount and the analytical blank. The analytical blank for DC 705 is several times greater than for Convalex 10. This is to be expected since silicone films are known to be very resistant to cleaning methods.

Table 3

ANALYTICAL TREATMENT OF OIL SAMPLES

Run	Station	Oil	Sample Weights (mg) Original	After First 3X Rinse	After Second 3X Rinse	Relationship Last to Original, %
156	1	Convalex 10	.020	.010	.010	50
160		Convalex 10	.020	.006	.003	15
174		Convalex 10	.032	.006	.003	10
166		Convalex 10	.019	.001	.001	5
157	2	DC 705	.047	.024	.020	42
161		DC 705	.063	.020	.020	32
172		DC 705	.078	.039	-	--
167		DC 705	.063	.020	.020	32
169	3	Convalex 10	.54	.006	.006	1
173		Convalex 10	.91	.016	.006	1
158	5	OS 124	.035	.018	.010	29
165	5	OS 124	.018	.009	.006	33
170	5	OS 124	.027	.003	-	--
171	5	OS 124	.039	.006	.003	8

E. Tests Under Standard Run Conditions

A number of runs made under standard run conditions are given in Table 4. Stations 1, 2, and 5 are running on the order of a magnitude lower than Stations 3 and 4. It will be recalled that Station 3 originally had a defective baffle which was subsequently replaced. Station 4 had previously been tested in the original work[2] where backstreaming values were approximately 1/4 present values. So far these two stations have shown very large values of light ends and types of pressure surges, which in the case of Station 3 were found to be hydrogen bursts. Stations 3 and 4 are presently being subjected to numerous regimes of oil conditioning, cleaning, new oil charges and different baffles.

Stations 1 and 5 with Convalex 10 and OS 124, respectively, are giving essentially the same very low values. Station 2 with DC 705 is giving somewhat higher values for total deposit weight. However, the latter, when corrected for the analytical blank, are essentially the same as those for Stations 1 and 5. These very low values are relatively independent of run length and correspond rather closely to the estimated amount in a monomolecular layer.

It appears from the results for these three stations operating under our standard conditions, that the same very low values can be obtained using widely different oils, baffles and pumps. The degree of oil conditioning and age of the oil has varied from two months

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*Technical Documentary Report No. AEDC-TDR-63-174, August 1963.

Table 4

BACKSTREAMING MEASUREMENT IN STANDARD RUNS

Run	Run Hours	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2: Total	Deposit Weight, mg/cm^2: Anal. Blank	Backstreaming Rate, $mg/(cm^2)(min)$: Total	Backstreaming Rate, $mg/(cm^2)(min)$: Less Anal. Blank
Station 1, PMC-1440A, Convalex 10, BC-61							
117	74	8×10^{-8}	6×10^{-9}	$.8 \times 10^{-4}$	-	$.024 \times 10^{-6}$	-
121	74	7×10^{-8}	9×10^{-9}	$.8 \times 10^{-4}$	-	$.025 \times 10^{-6}$	-
160	76	2×10^{-7}	9×10^{-9}	$.8 \times 10^{-4}$	$.1 \times 10^{-4}$	$.017 \times 10^{-6}$	$.015 \times 10^{-6}$
148	142	2×10^{-7}	4×10^{-9}	2.0×10^{-4}	-	$.032 \times 10^{-6}$	-
156	295	2×10^{-7}	1×10^{-9}	$.8 \times 10^{-4}$	$.04 \times 10^{-4}$	$.005 \times 10^{-6}$	$.004 \times 10^{-6}$
Station 2, HS6-1500, DC 705, BC-61							
118	74	6×10^{-9}	1×10^{-9}	1.9×10^{-4}	-	$.043 \times 10^{-6}$	-
122	74	5×10^{-9}	1×10^{-9}	2.4×10^{-4}	-	$.054 \times 10^{-6}$	-
135	74	3×10^{-9}	1×10^{-9}	3.2×10^{-4}	-	$.071 \times 10^{-6}$	-
161	74	9×10^{-9}	1×10^{-9}	2.5×10^{-4}	$.8 \times 10^{-4}$	$.057 \times 10^{-6}$	$.039 \times 10^{-6}$
149	145	4×10^{-9}	7×10^{-10}	3.2×10^{-4}	2.2×10^{-4}	$.036 \times 10^{-6}$	$.011 \times 10^{-6}$
157	293	5×10^{-9}	5×10^{-10}	1.9×10^{-4}	$.8 \times 10^{-4}$	$.011 \times 10^{-6}$	$.007 \times 10^{-6}$
Station 3, HS6-1500, Convalex 10, HN-6							
128	74	5×10^{-9}	2×10^{-9}	6.4×10^{-4}	-	$.14 \times 10^{-6}$	-
133	74	6×10^{-9}	2×10^{-9}	6.8×10^{-4}	-	$.15 \times 10^{-6}$	-
169	290	9×10^{-9}	2×10^{-9}	22.3×10^{-4}	$.2 \times 10^{-4}$	$.13 \times 10^{-6}$	$.13 \times 10^{-6}$
Station 4, HS6-1500, DC 705, Right Angle Elbow							
119	74	7×10^{-9}	2×10^{-9}	22.0×10^{-4}	-	$.49 \times 10^{-6}$	-
123	74	4×10^{-9}	3×10^{-9}	24.2×10^{-4}	-	$.54 \times 10^{-6}$	-
129	75	2×10^{-9}	9×10^{-10}	18.6×10^{-4}	-	$.41 \times 10^{-6}$	-
134	74	5×10^{-9}	1×10^{-9}	18.2×10^{-4}	-	$.41 \times 10^{-6}$	-
Station 5, HS6-1500, OS 124, Right Angle Elbow							
141	75	1×10^{-8}	5×10^{-9}	$.5 \times 10^{-4}$	-	$.015 \times 10^{-6}$	-
165	75	1×10^{-8}	4×10^{-9}	$.7 \times 10^{-4}$	$.3 \times 10^{-4}$	$.016 \times 10^{-6}$	$.010 \times 10^{-6}$
150	145	8×10^{-8}	8×10^{-8}	1.1×10^{-4}	-	$.013 \times 10^{-6}$	-
170	290	4×10^{-9}	2×10^{-9}	1.1×10^{-4}	$.1 \times 10^{-4}$	$.006 \times 10^{-6}$	$.006 \times 10^{-6}$

for Station 5 to three years for Station 2. Station 1, and previously Station 5, employed Convalex 10 with an accumulative run time of over 1 year.

The conditions of operation of these tests represent very good vacuum practice, conditions which are difficult or impractical to obtain in most vacuum chambers. It is believed that the conditions represent an optimum and that further reductions would not only be difficult to obtain but are far from actual practice. It is planned to use deviations from these optimum conditions to study the effects of various operating conditions such as air leaks, pulsing phenomena, etc.

E. Effect of Exposing Oil to Atmospheric Air

One of the first variables studied under these standardized run conditions was the effect of exposing room temperature oil to the atmosphere. It has been noted that new oil requires considerable degassing and usually undergoes severe frothing during initial operation. To exaggerate actual operating practice the conditioned oil from the two pumps (Stations 1 and 2) was drained and poured back into the pump to expose it to air. New oil cannot be tested since, without an extensive conditioning period of a month or so, backstreaming values are always extremely high. An additional run was also made where the pump oil was exposed to atmospheric pressure three successive times in the course of a single run. These data are given in Table 5. It can be seen that these tests had no appreciable effect on the amount of deposited oil.

Table 5

EFFECT OF EXPOSING PUMP OIL TO AIR AT ROOM TEMPERATURE

Run	Station	Oil	Remarks	Startup Rate, %	Bakeout Temp., °F	Run Hours	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2	Backstreaming Rate, $mg/(cm^2)(min)$
152	1	Convalex 10	Not aerated	50	None	72	-	-	1.4×10^{-4}	$.033 \times 10^{-6}$
130	1	Convalex 10	Aerated	50	None	74	8×10^{-8}	-	1.7×10^{-4}	$.052 \times 10^{-6}$
126	1	Convalex 10	Aerated	100	212	74	9×10^{-8}	9×10^{-9}	1.0×10^{-4}	$.030 \times 10^{-6}$
104	2	DC 705	Not aerated	50	None	70	3×10^{-8}	2×10^{-8}	5.0×10^{-4}	$.15 \times 10^{-6}$
107	2	DC 705	Not aerated	50	None	151	2×10^{-8}	2×10^{-8}	5.0×10^{-4}	$.056 \times 10^{-6}$
125	2	DC 705	Aerated	100	212	74	8×10^{-9}	2×10^{-9}	4.0×10^{-4}	$.088 \times 10^{-6}$
131	2	DC 705	Aerated	50	None	74	1×10^{-8}	2×10^{-9}	3.5×10^{-4}	$.078 \times 10^{-6}$
112	2	DC 705	Three starts	50	None	146	7×10^{-9}	2×10^{-9}	4.0×10^{-4}	$.045 \times 10^{-6}$

III. EXPERIMENTAL WORK - TURBO-MOLECULAR PUMP

Three turbo-molecular pumps, Model 3102A, 140 liter/min capacity, have been received from the Welch Scientific Company. Collection plates have been designed and fabricated. Current work involves connecting the pumps to electrical power and the baffle cooling system. No backstreaming data are available as yet.

IV. DISCUSSION

During this report period two factors have become apparent: (1) under optimum conditions the backstreaming of three systems using different pumps, baffles, and oils has been approximately 0.02×10^{-6} mg/(cm^2)(min) for standard run conditions of 70 hours and (2) the amount of oil on the collection plate is not greatly influenced by run time.

It is believed that the systems are operating under idealized conditions and, under these conditions, there is very little difference in pumps, oils, or baffles. It would appear that the difference in the systems will be apparent when operating under less-than-ideal conditions. The next effort should involve a study of each of the systems under conditions that more nearly simulate the vacuum practice of the many laboratories conducting space simulation work.

V. FUTURE WORK

A. Diffusion Pump

The following parameters which are representative of operating conditions will be studied with regards to their effect on backstreaming contamination.

1. Moist air leak into fine vacuum.
2. Moist air leak into fore line.
3. Evaluation of HN-6 (NRC) baffle.
4. Evaluation of series 251 Cryosorb cold trap Granville-Phillips baffle.
5. Bleed of light gas (helium) into fore line.

In addition, the effect of a side stream stripper on a station containing an oil which is giving very large backstreaming values will be investigated.

B. Turbo-Molecular Pumps

These pumps will be installed and initial tests conducted.

Respectfully submitted,

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